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| <p>(54) Title: EFFERVESCENCE COMPONENT</p> <p>(57) Abstract</p> <p>The invention provides an effervescence particle comprising an anhydrous perborate salt intimately mixed with a diluent, with the proviso that the diluent is not sodium perborate monohydrate. The invention also relates to processes for making the particle and to detergent compositions comprising such a particle. The invention also relates to the use of such an effervescence particle in a detergent composition for improvement of the dispensing or dissolution of the detergent composition into the washing water or for improvement of the delivery of detergent actives of the composition to the wash or for improved sudsing.</p>   |           |  |

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## **Effervescence Component**

### **Technical field**

This invention relates to a effervescence particle and detergent compositions comprising the particle.

### **Background to the Invention**

In recent years, detergent manufacturers have recognised as one of the problems encountered by consumers, that the detergent products do not always dispense or dissolve satisfactory in the washing water. This results in an inefficient delivery of the detergent actives to the wash, in lump gelling of the product in the washing machine and in deposition of product unto the fabric, which may result in fabric damage.

Various techniques have been introduced by detergent manufactures to improve the dispensing and dissolution of detergent products, including effervescence systems containing an acid and a carbonate salt, which form carbon dioxide-gas upon contact with water. In particular citric acid has been disclosed in various documents.

The inventors have found that a problem relating to these effervescence systems is that the presence of the acid lowers the pH of the washing water, which may not be desirable because certain detergent ingredients require an alkaline environment for maximum performance.

Another problem relating to these effervescence systems is that the acid does not just reacts with the carbonate salt upon contact with water in the wash, as required, but that the acid may react with other ingredients or with moisture during storage. Hereby, the effervescence is not delivered to its full potential. Furthermore, formulation detergents

with acids such as citric acid is expensive. Therefore, there is a need to provide improved effervescence systems.

The inventors have now found that improved effervescence can be achieved when anhydrous perborate is used.

Anhydrous perborate has been mentioned as potential effervescence system, which upon contact with water provides oxygen gas. For example US 4,4772,412 discloses a process for making a anhydrous perborate which can be used in liquid detergents. It mentions that the anhydrous perborate is unstable and losses performance when exposed to temperature increases or moisture. The inventors have found that this anhydrous perborate effervescence material is not suitable for use in most detergent products, in particular granular detergents.

Another problem with this material is that it may only be suitable of compositions which allow the presence of bleaching species, i.e. perborate monohydrate.

The inventors have now found that a stable effervescence component comprising anhydrous perborate, can be obtained when the anhydrous perborate is intimately mixed with a diluent, prior to addition to the detergent composition. The diluent can be intimately mixed with a hydrated perborate salt, prior to a drying step to form an anhydrous perborate salt, and/ or the diluent can be intimately mixed with the anhydrous perborate salt. These components are stable during manufacturing of the component and the detergent compositions and also upon storage. It has been found that the component of the invention provide improved effervescence and dispensing and dissolving of the product comprising the component. Furthermore, the anhydrous perborate provides an alkaline environment, which ensures an better performance of certain detergent ingredients. Furthermore, they have found that no perborate monohydrate or no high levels of perborate monohydrate needs to be present in the particle of the invention, which makes the particle more weight and cost efficient and moreover which makes the particle suitable for nil-bleach compositions.

As an additional benefit, the inventors have found that when the diluent comprises a surface active compound, an anhydrous perborate-containing component can be obtained, which produces effervescence and foaming or sudsing. Hereby, a controlled, rapid, improved sudsing and/ or foaming is achieved.

#### Summary of the invention

The invention provides an effervescence particle comprising an anhydrous perborate salt intimately mixed with a diluent, with the proviso that the diluent is not sodium perborate monohydrate. The invention also relates to processes for making the particle and to detergent compositions comprising such a particle.

The invention also relates to the use of such an effervescence particle in a detergent composition for improvement of the dispensing or dissolution of the detergent composition into the washing water or for improvement of the delivery of detergent actives of the composition to the wash or for improved sudsing.

#### Detailed description of the invention

The component of the invention comprises a diluent and an anhydrous perborate salt, which are intimately mixed. When used herein, 'intimately mixing/ mixed' or 'intimate mixture' means for the purpose of the invention that components of the foaming component are substantially homogeneously divided in the particle.

The particle of the invention is preferably substantially anhydrous. When used herein, 'substantially anhydrous' means that no more than 3% by weight of free moisture is present, preferably no more than 2%, even more preferably no more than 1% and most preferably no more than 0.75% or even 0.5% by weight. The free moisture content as

used herein, can be determined by placing 5 grams of the substantially anhydrous component in a petri dish and placing this petri dish in a convection oven at 50°C for 2 hours, and subsequently measuring the weight loss, due to water evaporation.

The intimate mixture of the components of the particle can be obtained by any process involving the mixing of the components, which can be part of a tableting process, extrusion process and agglomeration processes. When the particle comprises more than, the diluents are preferably premixed.

The component of the invention may be obtainable by a process comprising the steps of

- a) obtaining an anhydrous perborate salt;
- b) in a mixing step, intimately mixing the anhydrous perborate salt with a diluent.

Step a) may comprise the step of dehydration of a hydrated perborate salt to obtain an anhydrous perborate salt, optionally containing a hydrated perborate salt. If such a mixture of borate salts is present, the hydrated perborate salt is preferably less than 15%, preferably less than 10% or even 5% by weight of the total amount of perborate salt.

In step b), it may be preferred that the diluent or component thereof is present in liquid or liquified form. Such a liquid or liquefied diluent is preferably obtained by 1) formation of a melt of a diluent component, or 2) formation of a non-aqueous solution of the diluent component.

In 1), the diluent component and the anhydrous perborate salt are preferably intimately mixed at a temperature above the melting point of the diluent component, and the mixture solidifies on ambient temperature. Preferably, this process is performed at a temperature between 40°C and 200°C, or even 140°C or even 120°C more preferably between 40°C and 80°C.

In 2), the anhydrous perborate salt and the diluent present in a non-aqueous solution are intimately mixed, whereafter the solvent is dried.

The particle of the invention may alternatively be obtainable by a process comprising the steps of

- a) in a mixing step, intimately mixing a hydrated perborate salt, preferably a perborate monohydrate salt, with a diluent to obtain an intimate mixture;
- b) dehydration of the intimate mixture or the product of step a);
- c) optionally intimately mixing the product of step b) with a diluent.

The mixing steps as described herein may comprise the step of mixing the diluent and the anhydrous perborate salt or the diluent and the hydrate perborate salt, and then submitting the resulting mixture to an agglomeration step, pressure agglomeration step or compaction step, or an extrusion step.

The particle preferably comprises the anhydrous perborate salt at a level of from 1% to 80%, more preferably from 2% to 65%, even more preferably from 5% to 50% by weight of the particle. The particle preferably comprises the diluent at a level of from 20% to 99%, more preferably from 35% to 98%, even more preferably from 95% to 50% by weight of the particle.

The particle preferably has a particle size between 10 microns and 2 cm. The particle is preferably such that 80% by weight of the particles has an particle size of more than 75 microns (more than 80% by weight of the particles on Tyler sieve mesh 200) and less than 10% by weight of the particles has a particle size of more than 0.5 cm; preferably 80% by weight of the particles has an particle size of more than 100 microns (80% by weight on Tyler sieve mesh 100) and less than 10% by weight of the particles has a particle size of more than 0.1 cm; or more preferably 80% by weight of the particles has an particle size of more than 150 microns and less than 10% by weight of the particles has a particle size of more than 5000 microns; it may be preferred that the particles

have an average particle size of from 200 microns to 3000 microns, or even from 710 microns (on Tyler mesh sieve 24) to 1180 microns (through Tyler mesh sieve 14).

It may be preferred that the density of the particle is from 400 g/litre to 1200 g/litre, more preferably from 500 g/litre to 900g/litre.

The particle may be coated with any suitable coating agent which makes the particle less hygroscopic, whilst still allowing dissolution of the particle in the washing water.

Preferred coating agents may be, but are not limited to, paraffin oil, wax, nonionic surfactants, fatty acids, polymeric or oligomeric organic compounds including polyethylene glycols, polypropylene glycols and polymers or oligomers of acrylic and/or maleic acid or salts thereof. The coating agent may preferably be applied to the particle at a level of from 2% to 60%, more preferably from 5% to 20% or even to 15%.

#### Anhydrous perborate salts

The anhydrous perborate salt or oxoborate salt is preferably an anhydrous sodium or potassium perborate. It may be preferred that the direct product of the dehydration of a hydrated perborate salt, preferably sodium or potassium monohydrate, is incorporated in the particle of the invention. Then, depending on the conditions of the dehydration or drying process, e.g. the temperature, vacuum applied and air moisture content, certain amounts of hydrated perborate salt may be present in the product and in the particle of the invention.

However, it may be preferred to have only small amount of hydrated perborate present, e.g. up to 50% or more preferably up to 30% or even up to 20%, and it may be preferred that less than 15% or even less than 10% or even less than 5% by weight of the total amount of perborate salt is sodium perborate monohydrate or any hydrated perborate salt.

#### Diluent



The precise nature of the diluent and levels of incorporation thereof will depend on the application of the particle and the physical form of the particle. Preferably, the diluent is a detergent ingredient, which can function as stabiliser of the anhydrous perborate salt, whilst also performing a secondary function in the wash.

The diluent, preferably when intimately mixed with the anhydrous perborate salt, may comprise preferably a compound which has a melting point above 30°C, more preferably above 40°C, more preferably from 40°C to 200°C or to 140°C or even to 120°C or even 80°C. This is in particular to ensure that the diluent is solid under normal storage conditions, whilst readily forming a melt above the preferred melting point, depending on the application of the composition of the invention. In this embodiment, the diluent preferably comprises a polymeric organic compound or a anionic or nonionic surface active compound, preferably selected from the group comprising anionic alky sulphates or sulphonates, nonionic alkoxyated amides, alkyl esters of fatty acids, or alkoxyated alcohols, including polyethylene and/or propylene glycols, polymeric or oligomeric (poly) carboxylic acids or salts thereof, including polymeric or oligomeric acrylic/ maleic acids and salts thereof, alkoxyated fatty acid amides and alkoxyated alcoholamides, including ethanolamides and specific nonionic surfactants, including (polyhydroxy) fatty acid amides, alkoxyated alcohol surfactants, alkyl esters of fatty acids and specific alkyl polysaccherides surfactant or mixtures of any of these nonionics compounds and anionic compounds, as described herein.

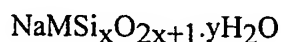
Highly may be one or more nonionic surfactant, selected from the group of nonionic alkoxyated surfactants, including alkoxyated alcohol surfactants, polyhydroxy fattyacid amide surfactants, (alkoxyated) fatty acid amide surfactants and alkylpolysaccharide surfactants, or mixtures thereof, as described herein after.

The diluent, in particular when intimately mixed with the hydrated perborate salt, may preferably comprises an organic or inorganic salt or acid. Preferably, the inorganic salts are silicate, sulphate, carbonate, borate (not being a perborate), phosphate, or halide,

preferably chloride, preferably the sodium, potassium or optionally calcium or magnesium salts thereof.

Suitable examples of phosphate salts are builders such as the alkali metal tripolyphosphates, sodium, potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from about 6 to 21, and salts of phytic acid.

Preferred diluents herein may comprise silicates, including amorphous silicates, crystalline layered silicates and aluminosilicates, such as zeolites. Synthetic aluminosilicate ion exchange materials for use herein are those available under the designations Zeolite A, Zeolite B, Zeolite P, Zeolite X, Zeolite HS and mixtures thereof. Preferred crystalline layered silicates for use herein have the general formula



wherein M is sodium or hydrogen, x is a number from 1.9 to 4 and y is a number from 0 to 20. Crystalline layered sodium silicates of this type are disclosed in EP-A-0164514 and methods for their preparation are disclosed in DE-A-3417649 and DE-A-3742043. Herein, x in the general formula above preferably has a value of 2, 3 or 4 and is preferably 2. The most preferred material is  $\delta\text{-Na}_2\text{Si}_2\text{O}_5$ , available from Hoechst AG as NaSKS-6.

As described above, also preferred diluents may be polymeric, oligomeric or monomeric (poly) carboxylic acid or salt thereof.

Suitable carboxylates containing one carboxy group include the water soluble salts of lactic acid, glycolic acid and ether derivatives thereof. Polycarboxylates containing two carboxy groups include the water-soluble salts of succinic acid, malonic acid,

(ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the ether carboxylates and the sulfinyl carboxylates.

Polycarboxylates containing three carboxy groups include, in particular, water-soluble citrates, aconitrates and citraconates as well as succinate derivatives such as the carboxymethyloxysuccinates described in British Patent No. 1,379,241, lactoxysuccinates described in British Patent No. 1,389,732, and aminosuccinates described in Netherlands Application 7205873, and the oxypolycarboxylate materials such as 2-oxa-1,1,3-propane tricarboxylates described in British Patent No. 1,387,447. Polycarboxylates containing four carboxy groups include oxydisuccinates disclosed in British Patent No. 1,261,829, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates. Polycarboxylates containing sulfo substituents include the sulfosuccinate derivatives disclosed in British Patent Nos. 1,398,421 and 1,398,422 and in U.S. Patent No. 3,936,448, and the sulfonated pyrolysed citrates described in British Patent No. 1,439,000. Preferred polycarboxylates are hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates. The parent acids of the monomeric or oligomeric polycarboxylates or mixtures thereof with their salts, e.g. citric acid or citrate/citric acid mixtures are also contemplated as useful herein.

It may be preferred that the particle of the invention comprises one or more surfactants, as described above and more detail herein after. Namely, it has been found that the presence of a surfactant in the particle of the invention, can provide excellent foaming or sudsing without agitation. The surfactant used in the particle of the invention are preferably substantially anhydrous.

### Compositions

The particle of the invention is particularly useful in non-aqueous liquid compositions or preferably solid compositions. The particle may be present as a separate component or the particle can be present as a part of a component of the solid or non-aqueous liquid composition. The solid cleaning compositions are preferably solid laundry or dish washing compositions, preferably in the form of flakes or pastilles, more preferably in

the form of granules or extrudates or tablets It may be preferred that the compositions have a density of at least 400g/litre, more preferably at least 500g/litre.

The particle of the invention for use in cleaning compositions, and/ or the cleaning compositions preferably contain one or more additional detergent components selected from surfactants, bleaches, builders, chelants, (additional) alkalinity sources, organic polymeric compounds, effervescence systems, enzymes, suds suppressors, lime soap dispersants, brighteners, soil suspension and anti-redeposition agents and corrosion inhibitors.

Highly preferred additional deterative actives or ingredients are cationic, nonionic and anionic surfactants, as described above, suds suppressing systems, brighteners, and bleaching compounds, including perhydrate bleaches but preferably bleach activators, as described hereinafter.

#### Surfactants

The particle of the invention, as described above, and/ or the compositions comprising the particle preferably comprise one or more surfactants, in particularly laundry and dish washing compositions herein.

#### Nonionic alkoxyated surfactant

Essentially any alkoxyated nonionic surfactants can also be comprised in the particles or compositions herein. The ethoxyated and propoxyated nonionic surfactants are preferred. Preferred alkoxyated surfactants can be selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxyated alcohols, nonionic ethoxyated/propoxyated fatty alcohols, nonionic ethoxylate/propoxylate condensates with propylene glycol, and the nonionic ethoxylate condensation products with propylene oxide/ethylene diamine adducts.

Highly preferred are nonionic alkoxyated alcohol surfactants, being the condensation products of aliphatic alcohols with from 1 to 75, up to 50 moles, preferably 1 to 15 moles of alkylene oxide, particularly ethylene oxide and/or propylene oxide, are highly preferred nonionic surfactant comprised in the anhydrous component of the particles of the invention. The alkyl chain of the aliphatic alcohol can either be straight or branched, primary or secondary, and generally contains from 6 to 22 carbon atoms. Particularly preferred are the condensation products of alcohols having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

#### Nonionic polyhydroxy fatty acid amide surfactant

Polyhydroxy fatty acid amides are highly preferred nonionic surfactant comprised in the particle or compositions herein, in particular those having the structural formula  $R^2CONR^1Z$  wherein :  $R^1$  is H,  $C_{1-18}$ , preferably  $C_1-C_4$  hydrocarbyl, 2-hydroxy ethyl, 2-hydroxy propyl, ethoxy, propoxy, or a mixture thereof, preferable  $C_1-C_4$  alkyl, more preferably  $C_1$  or  $C_2$  alkyl, most preferably  $C_1$  alkyl (i.e., methyl); and  $R^2$  is a  $C_5-C_{31}$  hydrocarbyl, preferably straight-chain  $C_5-C_{19}$  or  $C_7-C_{19}$  alkyl or alkenyl, more preferably straight-chain  $C_9-C_{17}$  alkyl or alkenyl, most preferably straight-chain  $C_{11}-C_{17}$  alkyl or alkenyl, or mixture thereof; and Z is a polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 3 hydroxyls directly connected to the chain, or an alkoxyated derivative (preferably ethoxylated or propoxylated) thereof. Z preferably will be derived from a reducing sugar in a reductive amination reaction; more preferably Z is a glycityl.

A highly preferred nonionic polyhydroxy fatty acid amide surfactant for use herein is a  $C_{12}-C_{14}$ , a  $C_{15}-C_{17}$  and/or  $C_{16}-C_{18}$  alkyl N-methyl glucamide.

It may be particularly preferred that the particle or the compositions herein comprises a mixture of a C<sub>12</sub>-C<sub>18</sub> alkyl N-methyl glucamide and a condensation products of a alcohol having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

The polyhydroxy fatty acid amide can be prepared by any suitable process. One particularly preferred process is described in detail in WO 9206984. A product comprising about 95% by weight polyhydroxy fatty acid amide, low levels of undesired impurities such as fatty acid esters and cyclic amides, and which is molten typically above about 80°C, can be made by this process

#### Nonionic fatty acid amide surfactant

Fatty acid amide surfactants or alkoxylated fatty acid amides can also be comprised in the particle of the invention or compositions herein. They include those having the formula:  $R^6CON(R^7)(R^8)$  wherein  $R^6$  is an alkyl group containing from 7 to 21, preferably from 9 to 17 carbon or even 12 to 14 carbon atoms and  $R^7$  and  $R^8$  are each individually selected from the group consisting of hydrogen, C<sub>1</sub>-C<sub>4</sub> alkyl, C<sub>1</sub>-C<sub>4</sub> hydroxyalkyl, and  $-(C_2H_4O)_xH$ , where x is in the range of from 1 to 11, preferably 1 to 7, more preferably from 1-5, whereby it may be preferred that  $R^7$  is different to  $R^8$ , one having a smaller alkoxylation number than the other.

#### Nonionic alkyl esters of fatty acid surfactant

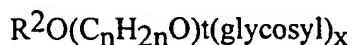
Alkyl esters of fatty acids can also be comprised in the particle of the invention or compositions herein. They include those having the formula:  $R^9COO(R^{10})$  wherein  $R^9$  is an alkyl group containing from 7 to 21, preferably from 9 to 17 carbon or even 11 to 13 carbon atoms and  $R^{10}$  is a C<sub>1</sub>-C<sub>4</sub> alkyl, C<sub>1</sub>-C<sub>4</sub> hydroxyalkyl, or  $-(C_2H_4O)_xH$ , where

x is in the range of from 1 to 11, preferably 1 to 7, more preferably from 1-5, whereby it may be preferred that R<sup>10</sup> is a methyl or ethyl group.

#### Nonionic alkylpolysaccharide surfactant

Alkylpolysaccharides can also be comprised in the particles or compositions herein, such as those disclosed in U.S. Patent 4,565,647, Llenado, issued January 21, 1986, having a hydrophobic group containing from 6 to 30 carbon atoms and a polysaccharide, e.g., a polyglycoside, hydrophilic group containing from 1.3 to 10 saccharide units.

Preferred alkylpolyglycosides have the formula



wherein R<sup>2</sup> is selected from the group consisting of alkyl, alkylphenyl, hydroxyalkyl, hydroxyalkylphenyl, and mixtures thereof in which the alkyl groups contain from 10 to 18 carbon atoms; n is 2 or 3; t is from 0 to 10, and x is from 1.3 to 8. The glycosyl is preferably derived from glucose.

#### Anionic surfactant

The particle and/ or the composition herein may comprises one or more anionic surfactants. Any anionic surfactant useful for deterative purposes is suitable. Examples include salts (including, for example, sodium, potassium, ammonium, and substituted ammonium salts such as mono-, di- and triethanolamine salts) of the anionic sulfate, sulfonate, carboxylate and sarcosinate surfactants. Anionic sulfate surfactants are preferred.

Other anionic surfactants include the isethionates such as the acyl isethionates, N-acyl taurates, fatty acid amides of methyl tauride, alkyl succinates and sulfosuccinates,

monoesters of sulfosuccinate (especially saturated and unsaturated  $C_{12}$ - $C_{18}$  monoesters) diesters of sulfosuccinate (especially saturated and unsaturated  $C_6$ - $C_{14}$  diesters), N-acyl sarcosinates. Resin acids and hydrogenated resin acids are also suitable, such as rosin, hydrogenated rosin, and resin acids and hydrogenated resin acids present in or derived from tallow oil.

The anionic surfactant may preferably be present at a level of 1% to 90% by weight of the composition, preferably at a level of from 5% to 60%, and preferably of from 8% to 50% by weight.

#### Anionic sulfate surfactant

Anionic sulfate surfactants suitable for use herein include the linear and branched primary and secondary alkyl sulfates, alkyl ethoxysulfates, fatty oleoyl glycerol sulfates, alkyl phenol ethylene oxide ether sulfates, the  $C_5$ - $C_{17}$  acyl-N-( $C_1$ - $C_4$  alkyl) and -N-( $C_1$ - $C_2$  hydroxyalkyl) glucamine sulfates, and sulfates of alkylpolysaccharides such as the sulfates of alkylpolyglucoside (the nonionic nonsulfated compounds being described herein).

Alkyl sulfate surfactants are preferably selected from the linear and branched primary  $C_9$ - $C_{22}$  alkyl sulfates, more preferably the  $C_{11}$ - $C_{15}$  branched chain alkyl sulfates and the  $C_{12}$ - $C_{14}$  linear chain alkyl sulfates.

Alkyl ethoxysulfate surfactants are preferably selected from the group consisting of the  $C_{10}$ - $C_{18}$  alkyl sulfates which have been ethoxylated with from 0.5 to 50 moles of ethylene oxide per molecule. More preferably, the alkyl ethoxysulfate surfactant is a  $C_{11}$ - $C_{18}$ , most preferably  $C_{11}$ - $C_{15}$  alkyl sulfate which has been ethoxylated with from 0.5 to 7, preferably from 1 to 5, moles of ethylene oxide per molecule.



Anionic sulfonate surfactant

Anionic sulfonate surfactants suitable for use herein include the salts of C<sub>5</sub>-C<sub>20</sub> linear or branched alkylbenzene sulfonates, alkyl ester sulfonates, in particular methyl ester sulphonates, C<sub>6</sub>-C<sub>22</sub> primary or secondary alkane sulfonates, C<sub>6</sub>-C<sub>24</sub> olefin sulfonates, sulfonated polycarboxylic acids, alkyl glycerol sulfonates, fatty acyl glycerol sulfonates, fatty oleyl glycerol sulfonates, and any mixtures thereof.

Anionic carboxylate surfactant

Suitable anionic carboxylate surfactants include the alkyl ethoxy carboxylates, the alkyl polyethoxy polycarboxylate surfactants and the soaps ('alkyl carboxyls'), especially certain secondary soaps as described herein.

Suitable alkyl ethoxy carboxylates include those with the formula  $RO(CH_2CH_2O)_xCH_2COO^-M^+$  wherein R is a C<sub>6</sub> to C<sub>18</sub> alkyl group, x ranges from 0 to 10, and the ethoxylate distribution is such that, on a weight basis, the amount of material where x is 0 is less than 20 % and M is a cation. Suitable alkyl polyethoxy polycarboxylate surfactants include those having the formula  $RO-(CHR_1-CHR_2-O)_x-R_3$  wherein R is a C<sub>6</sub> to C<sub>18</sub> alkyl group, x is from 1 to 25, R<sub>1</sub> and R<sub>2</sub> are selected from the group consisting of hydrogen, methyl acid radical, succinic acid radical, hydroxysuccinic acid radical, and mixtures thereof, and R<sub>3</sub> is selected from the group consisting of hydrogen, substituted or unsubstituted hydrocarbon having between 1 and 8 carbon atoms, and mixtures thereof.

Suitable soap surfactants include the secondary soap surfactants which contain a carboxyl unit connected to a secondary carbon. Preferred secondary soap surfactants for use herein are water-soluble members selected from the group consisting of the water-

soluble salts of 2-methyl-1-undecanoic acid, 2-ethyl-1-decanoic acid, 2-propyl-1-nonanoic acid, 2-butyl-1-octanoic acid and 2-pentyl-1-heptanoic acid. Certain soaps may also be included as suds suppressors.

#### Alkali metal sarcosinate surfactant

Other suitable anionic surfactants are the alkali metal sarcosinates of formula  $R-CON(R^1)CH_2COOM$ , wherein R is a  $C_5$ - $C_{17}$  linear or branched alkyl or alkenyl group,  $R^1$  is a  $C_1$ - $C_4$  alkyl group and M is an alkali metal ion. Preferred examples are the myristyl and oleoyl methyl sarcosinates in the form of their sodium salts.

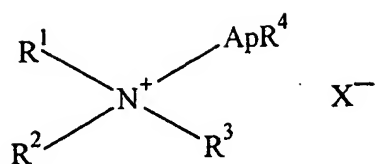
#### Cationic surfactant

A cationic surfactant may be comprised in the particle and/ or the composition herein, preferably be present at a level of from 0.5% to 80% by weight of the component, more preferably from 1% to 60%, most preferably from 3% to 50% by weight of the component.

Preferably the cationic surfactant is selected from the group consisting of cationic ester surfactants, cationic mono-alkoxylated amine surfactants, cationic bis-alkoxylated amine surfactants and mixtures thereof.

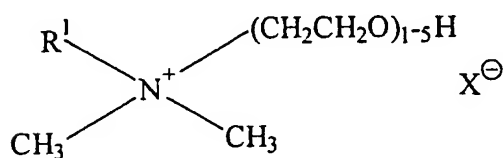
#### Cationic mono-alkoxylated amine surfactants

The optional cationic mono-alkoxylated amine surfactant for use herein, has the general formula:



wherein  $\text{R}^1$  is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms;  $\text{R}^2$  and  $\text{R}^3$  are each independently alkyl groups containing from one to about three carbon atoms, preferably methyl;  $\text{R}^4$  is selected from hydrogen (preferred), methyl and ethyl,  $\text{X}^-$  is an anion such as chloride, bromide, methylsulfate, sulfate, or the like, to provide electrical neutrality; A is selected from  $\text{C}_1$ - $\text{C}_4$  alkoxy, especially ethoxy (i.e.,  $-\text{CH}_2\text{CH}_2\text{O}-$ ), propoxy, butoxy and mixtures thereof; and p is from 1 to about 30, preferably 1 to about 15, most preferably 1 to about 8.

Highly preferred cationic mono-alkoxylated amine surfactants for use herein are of the formula

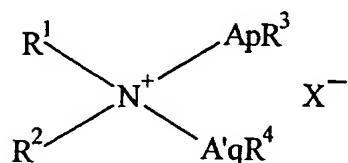


wherein  $\text{R}^1$  is  $\text{C}_6$ - $\text{C}_{18}$  hydrocarbyl and mixtures thereof, preferably  $\text{C}_6$ - $\text{C}_{14}$ , especially  $\text{C}_6$ - $\text{C}_{14}$  alkyl, preferably  $\text{C}_{12}$  and  $\text{C}_{14}$  alkyl, and X is any convenient anion to provide charge balance, preferably chloride or bromide.

As noted, compounds of the foregoing type include those wherein the ethoxy  $(\text{CH}_2\text{CH}_2\text{O})$  units (EO) are replaced by butoxy, isopropoxy  $[\text{CH}(\text{CH}_3)\text{CH}_2\text{O}]$  and  $[\text{CH}_2\text{CH}(\text{CH}_3)\text{O}]$  units (i-Pr) or n-propoxy units (Pr), or mixtures of EO and/or Pr and/or i-Pr units.

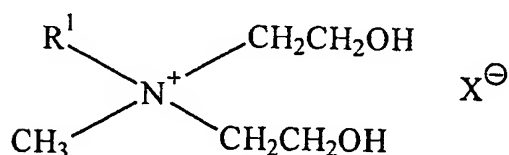
Cationic bis-alkoxylated amine surfactant

The cationic bis-alkoxylated amine surfactant for use herein, has the general formula:



wherein  $\text{R}^1$  is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms, more preferably 6 to about 11, most preferably from about 8 to about 10 carbon atoms;  $\text{R}^2$  is an alkyl group containing from one to three carbon atoms, preferably methyl;  $\text{R}^3$  and  $\text{R}^4$  can vary independently and are selected from hydrogen (preferred), methyl and ethyl,  $\text{X}^-$  is an anion such as chloride, bromide, methylsulfate, sulfate, or the like, sufficient to provide electrical neutrality. A and A' can vary independently and are each selected from  $\text{C}_1$ - $\text{C}_4$  alkoxy, especially ethoxy, (i.e.,  $-\text{CH}_2\text{CH}_2\text{O}-$ ), propoxy, butoxy and mixtures thereof; p is from 1 to about 30, preferably 1 to about 4 and q is from 1 to about 30, preferably 1 to about 4, and most preferably both p and q are 1.

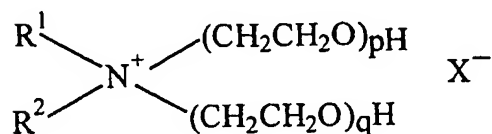
Highly preferred cationic bis-alkoxylated amine surfactants for use herein are of the formula



wherein  $\text{R}^1$  is  $\text{C}_6$ - $\text{C}_{18}$  hydrocarbyl and mixtures thereof, preferably  $\text{C}_6$ ,  $\text{C}_8$ ,  $\text{C}_{10}$ ,  $\text{C}_{12}$ ,  $\text{C}_{14}$  alkyl and mixtures thereof. X is any convenient anion to provide charge balance, preferably chloride. With reference to the general cationic bis-alkoxylated amine

structure noted above, since in a preferred compound  $R^1$  is derived from (coconut)  $C_{12}$ - $C_{14}$  alkyl fraction fatty acids,  $R^2$  is methyl and  $ApR^3$  and  $A'qR^4$  are each monoethoxy.

Other cationic bis-alkoxylated amine surfactants useful herein include compounds of the formula:



wherein  $R^1$  is  $C_6$ - $C_{18}$  hydrocarbyl, preferably  $C_6$ - $C_{14}$  alkyl, independently  $p$  is 1 to about 3 and  $q$  is 1 to about 3,  $R^2$  is  $C_1$ - $C_3$  alkyl, preferably methyl, and  $X$  is an anion, especially chloride or bromide.

Other compounds of the foregoing type include those wherein the ethoxy ( $CH_2CH_2O$ ) units (EO) are replaced by butoxy (Bu) isopropoxy [ $CH(CH_3)CH_2O$ ] and [ $CH_2CH(CH_3O)$ ] units (i-Pr) or n-propoxy units (Pr), or mixtures of EO and/or Pr and/or i-Pr units.

#### Amphoteric surfactant

Suitable amphoteric surfactants for use herein include the amine oxide surfactants and the alkyl amphocarboxylic acids.

Suitable amine oxides include those compounds having the formula  $R^3(OR^4)_xN^0(R^5)_2$  wherein  $R^3$  is selected from an alkyl, hydroxyalkyl, acylamidopropoyl and alkyl phenyl group, or mixtures thereof, containing from 8 to 26 carbon atoms;  $R^4$  is an alkylene or

hydroxyalkylene group containing from 2 to 3 carbon atoms, or mixtures thereof; x is from 0 to 5, preferably from 0 to 3; and each  $R^5$  is an alkyl or hydroxyalkyl group containing from 1 to 3, or a polyethylene oxide group containing from 1 to 3 ethylene oxide groups. Preferred are  $C_{10}$ - $C_{18}$  alkyl dimethylamine oxide, and  $C_{10-18}$  acylamido alkyl dimethylamine oxide.

A suitable example of an alkyl aphodicarboxylic acid is Miranol(TM) C2M Conc. manufactured by Miranol, Inc., Dayton, NJ.

#### Zwitterionic surfactant

Zwitterionic surfactants can also be comprised in the surface active component of the composition of the invention or the compositions containing the particle of the invention. These surfactants can be broadly described as derivatives of secondary and tertiary amines, derivatives of heterocyclic secondary and tertiary amines, or derivatives of quaternary ammonium, quaternary phosphonium or tertiary sulfonium compounds. Betaine and sultaine surfactants are exemplary zwitterionic surfactants for use herein.

Suitable betaines are those compounds having the formula  $R(R')_2N^+R^2COO^-$  wherein R is a  $C_6$ - $C_{18}$  hydrocarbyl group, each  $R^1$  is typically  $C_1$ - $C_3$  alkyl, and  $R^2$  is a  $C_1$ - $C_5$  hydrocarbyl group. Preferred betaines are  $C_{12-18}$  dimethyl-ammonio hexanoate and the  $C_{10-18}$  acylamidopropane (or ethane) dimethyl (or diethyl) betaines. Complex betaine surfactants are also suitable for use herein.

#### Suds suppressing system

The particle of the invention or the composition herein, in particular when comprising high foaming surfactant systems, can provide a very rapid formation of very stable foam.

However, it may be preferred that the foaming is limited or reduced at a certain moment during the washing process, for example to improve the drainage of the suds or foam from the washing process or the machine. Also, it may be preferred that the foaming is limited to the start of the contact with water (e.g. the beginning of the washing process), to provide effervescence, or improved dispensing or dissolving of the particle or the product comprising the particle. Therefore, it may be preferably comprise a suds suppressing system present at a level of from 0.01% to 15%, preferably from 0.05% to 10%, most preferably from 0.1% to 5% by weight of the composition or the component.

Suitable suds suppressing systems for use herein may comprise essentially any known antifoam compound, including, for example silicone antifoam compounds and 2-alkyl alcanol antifoam compounds.

By antifoam compound it is meant herein any compound or mixtures of compounds which act such as to depress the foaming or sudsing produced by a solution of a detergent composition.

Particularly preferred antifoam compounds for use herein are silicone antifoam compounds defined herein as any antifoam compound including a silicone component. Such silicone antifoam compounds also typically contain a silica component. The term "silicone" as used herein, and in general throughout the industry, encompasses a variety of relatively high molecular weight polymers containing siloxane units and hydrocarbyl group of various types. Preferred silicone antifoam compounds are the siloxanes, particularly the polydimethylsiloxanes having trimethylsilyl end blocking units.

Other suitable antifoam compounds include the monocarboxylic fatty acids and soluble salts thereof. These materials are described in US Patent 2,954,347, issued September 27, 1960 to Wayne St. John. The monocarboxylic fatty acids, and salts thereof, for use as suds suppressor typically have hydrocarbyl chains of 10 to 24 carbon atoms,

preferably 12 to 18 carbon atoms. Suitable salts include the alkali metal salts such as sodium, potassium, and lithium salts, and ammonium and alkanolammonium salts.

Other suitable antifoam compounds include, for example, high molecular weight fatty esters (e.g. fatty acid triglycerides), fatty acid esters of monovalent alcohols, aliphatic C<sub>18</sub>-C<sub>40</sub> ketones (e.g. stearone) N-alkylated amino triazines such as tri- to hexa-alkylmelamines or di- to tetra alkyl diamine chlortriazines formed as products of cyanuric chloride with two or three moles of a primary or secondary amine containing 1 to 24 carbon atoms, propylene oxide, bis stearic acid amide and monostearyl di-alkali metal (e.g. sodium, potassium, lithium) phosphates and phosphate esters.

A preferred suds suppressing system comprises

(a) antifoam compound, preferably silicone antifoam compound, most preferably a silicone antifoam compound comprising in combination

(i) polydimethyl siloxane, at a level of from 50% to 99%, preferably 75% to 95% by weight of the silicone antifoam compound; and

(ii) silica, at a level of from 1% to 50%, preferably 5% to 25% by weight of the silicone/silica antifoam compound;

wherein said silica/silicone antifoam compound is incorporated at a level of from 5% to 50%, preferably 10% to 40% by weight;

(b) a dispersant compound, most preferably comprising a silicone glycol rake copolymer with a polyoxyalkylene content of 72-78% and an ethylene oxide to propylene oxide ratio of from 1:0.9 to 1:1.1, at a level of from 0.5% to 10%, preferably 1% to 10% by weight; a particularly preferred silicone glycol rake



copolymer of this type is DCO544, commercially available from DOW Corning under the tradename DCO544;

- (c) an inert carrier fluid compound, most preferably comprising a C<sub>16</sub>-C<sub>18</sub> ethoxylated alcohol with a degree of ethoxylation of from 5 to 50, preferably 8 to 15, at a level of from 5% to 80%, preferably 10% to 70%, by weight;

A highly preferred particulate suds suppressing system is described in EP-A-0210731 and comprises a silicone antifoam compound and an organic carrier material having a melting point in the range 50°C to 85°C, wherein the organic carrier material comprises a monoester of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms. EP-A-0210721 discloses other preferred particulate suds suppressing systems wherein the organic carrier material is a fatty acid or alcohol having a carbon chain containing from 12 to 20 carbon atoms, or a mixture thereof, with a melting point of from 45°C to 80°C. Other highly preferred suds suppressing systems comprise a mixture of silicones or siloxanes with alumino silicates and polycarboxylate polymers.

#### Water-soluble builder compound

The particle or the cleaning compositions herein preferably contain a water-soluble builder compound, typically present at a level of from 1% to 80% by weight, preferably from 10% to 70% by weight, most preferably from 20% to 60% by weight of the composition or component.

Suitable water-soluble builder compounds include the water soluble monomeric polycarboxylates, or their acid forms, homo or copolymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxylic radicals separated from each other by not more than two carbon atoms, as described above, borates, phosphates, and mixtures of any of the foregoing.

Borate builders, as well as builders containing borate-forming materials that can produce borate under detergent storage or wash conditions are useful water-soluble builders herein.

Suitable examples of water-soluble phosphate builders are the alkali metal tripolyphosphates, sodium, potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from about 6 to 21, and salts of phytic acid.

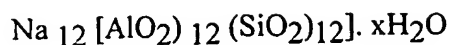
Partially soluble or insoluble builder compound

The particle or compositions containing the particle of the invention may contain a partially soluble or insoluble builder compound, typically present at a level of from 1% to 80% by weight, preferably from 10% to 70% by weight, most preferably from 20% to 60% weight of the composition or component

Examples of largely water insoluble builders include the sodium aluminosilicates.

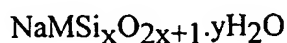
Suitable aluminosilicate zeolites have the unit cell formula  $\text{Na}_z[(\text{AlO}_2)_z(\text{SiO}_2)_y] \cdot x\text{H}_2\text{O}$  wherein z and y are at least 6; the molar ratio of z to y is from 1.0 to 0.5 and x is at least 5, preferably from 7.5 to 276, more preferably from 10 to 264. The aluminosilicate material are in hydrated form and are preferably crystalline, containing from 10% to 28%, more preferably from 18% to 22% water in bound form.

The aluminosilicate zeolites can be naturally occurring materials, but are preferably synthetically derived. Synthetic crystalline aluminosilicate ion exchange materials are available under the designations Zeolite A, Zeolite B, Zeolite P, Zeolite X, Zeolite HS and mixtures thereof. Zeolite A has the formula



wherein x is from 20 to 30, especially 27. Zeolite X has the formula  $\text{Na}_{46} [(\text{AlO}_2)_{86}(\text{SiO}_2)_{106}] \cdot 276 \text{H}_2\text{O}$ .

Preferred crystalline layered silicates for use herein have the general formula



wherein M is sodium or hydrogen, x is a number from 1.9 to 4 and y is a number from 0 to 20. Crystalline layered sodium silicates of this type are disclosed in EP-A-0164514 and methods for their preparation are disclosed in DE-A-3417649 and DE-A-3742043. Herein, x in the general formula above preferably has a value of 2, 3 or 4 and is preferably 2. The most preferred material is  $\delta\text{-Na}_2\text{Si}_2\text{O}_5$ , available from Hoechst AG as NaSKS-6.

#### Perhydrate bleaches

An preferred additional components of the particle of the invention or the composition herein, is a perhydrate bleach, such as metal perborates, metal percarbonates, particularly the sodium salts. Perborate can be mono or tetra hydrated. Sodium percarbonate has the formula corresponding to  $2\text{Na}_2\text{CO}_3 \cdot 3\text{H}_2\text{O}_2$ , and is available commercially as a crystalline solid.

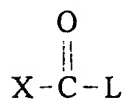
Potassium peroxymonopersulfate, sodium per is another optional inorganic perhydrate salt of use in the detergent compositions or foaming components.

#### Organic peroxyacid bleaching system

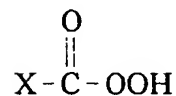
A preferred feature of the particle or compositions herein, is an organic peroxyacid bleaching system. In one preferred execution the bleaching system contains a hydrogen peroxide source and an organic peroxyacid bleach precursor compound. The production of the organic peroxyacid occurs by an in situ reaction of the precursor with a source of hydrogen peroxide. Preferred sources of hydrogen peroxide include inorganic perhydrate bleaches, such as the perborate bleach of the claimed invention. In an alternative preferred execution a preformed organic peroxyacid is incorporated directly into the composition. Compositions containing mixtures of a hydrogen peroxide source and organic peroxyacid precursor in combination with a preformed organic peroxyacid are also envisaged.

#### Peroxyacid bleach precursor

Peroxyacid bleach precursors are compounds which react with hydrogen peroxide in a perhydrolysis reaction to produce a peroxyacid. Generally peroxyacid bleach precursors may be represented as



where L is a leaving group and X is essentially any functionality, such that on perhydroloysis the structure of the peroxyacid produced is



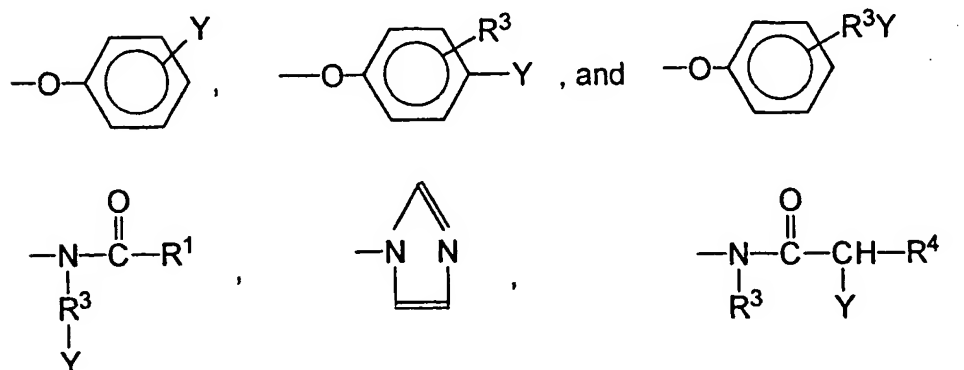
Peroxyacid bleach precursor compounds are preferably incorporated at a level of from 0.5% to 6% by weight, more preferably from 1% to 40 by weight, most preferably from 1.5% to 25 by weight of the compositions or component

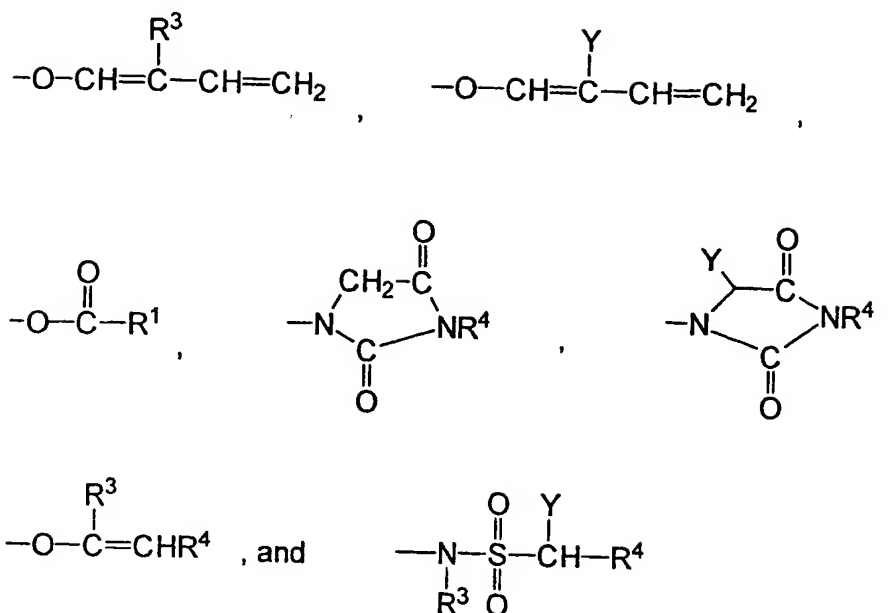
Suitable peroxyacid bleach precursor compounds typically contain one or more N- or O-acyl groups, which precursors can be selected from a wide range of classes. Suitable classes include anhydrides, esters, imides, lactams and acylated derivatives of imidazoles and oximes. Examples of useful materials within these classes are disclosed in GB-A-1586789. Suitable esters are disclosed in GB-A-836988, 864798, 1147871, 2143231 and EP-A-0170386.

### Leaving groups

The leaving group, hereinafter L group, must be sufficiently reactive for the perhydrolysis reaction to occur within the optimum time frame (e.g., a wash cycle). However, if L is too reactive, this activator will be difficult to stabilize for use in a bleaching composition.

Preferred L groups are selected from the group consisting of:





and mixtures thereof, wherein  $\text{R}^1$  is an alkyl, aryl, or alkaryl group containing from 1 to 14 carbon atoms,  $\text{R}^3$  is an alkyl chain containing from 1 to 8 carbon atoms,  $\text{R}^4$  is H or  $\text{R}^3$ , and Y is H or a solubilizing group. Any of  $\text{R}^1$ ,  $\text{R}^3$  and  $\text{R}^4$  may be substituted by essentially any functional group including, for example alkyl, hydroxy, alkoxy, halogen, amine, nitrosyl, amide and ammonium or alkyl ammonium groups.

The preferred solubilizing groups are  $-\text{SO}_3^- \text{M}^+$ ,  $-\text{CO}_2^- \text{M}^+$ ,  $-\text{SO}_4^- \text{M}^+$ ,  $-\text{N}^+(\text{R}^3)_4 \text{X}^-$  and  $\text{O}^<- \text{N}(\text{R}^3)_3$  and most preferably  $-\text{SO}_3^- \text{M}^+$  and  $-\text{CO}_2^- \text{M}^+$  wherein  $\text{R}^3$  is an alkyl chain containing from 1 to 4 carbon atoms, M is a cation which provides solubility to the bleach activator and X is an anion which provides solubility to the bleach activator. Preferably, M is an alkali metal, ammonium or substituted ammonium cation, with sodium and potassium being most preferred, and X is a halide, hydroxide, methylsulfate or acetate anion.

#### Alkyl percarboxylic acid bleach precursors

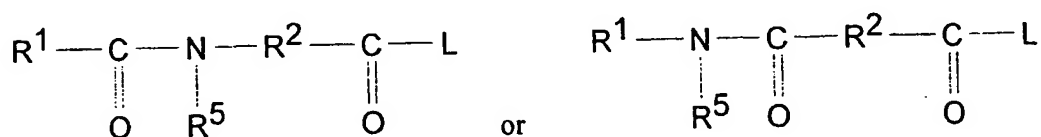
Alkyl percarboxylic acid bleach precursors form percarboxylic acids on perhydrolysis. Preferred precursors of this type provide peracetic acid on perhydrolysis.

Preferred alkyl percarboxylic precursor compounds of the imide type include the N-,N,N<sup>1</sup>N<sup>1</sup> tetra acetylated alkylene diamines wherein the alkylene group contains from 1 to 6 carbon atoms, particularly those compounds in which the alkylene group contains 1, 2 and 6 carbon atoms. Tetraacetyl ethylene diamine (TAED) is particularly preferred. The TAED is preferably not present in the agglomerated particle of the present invention, but preferably present in the detergent composition, comprising the particle.

Other preferred alkyl percarboxylic acid precursors include sodium 3,5,5-tri-methyl hexanoyloxybenzene sulfonate (iso-NOBS), sodium nonanoyloxybenzene sulfonate (NOBS), sodium acetoxybenzene sulfonate (ABS) and pentaacetyl glucose.

#### Amide substituted alkyl peroxyacid precursors

Amide substituted alkyl peroxyacid precursor compounds are suitable herein, including those of the following general formulae:



wherein R<sup>1</sup> is an alkyl group with from 1 to 14 carbon atoms, R<sup>2</sup> is an alkylene group containing from 1 to 14 carbon atoms, and R<sup>5</sup> is H or an alkyl group containing 1 to 10 carbon atoms and L can be essentially any leaving group. Amide substituted bleach activator compounds of this type are described in EP-A-0170386.

### Perbenzoic acid precursor

Perbenzoic acid precursor compounds provide perbenzoic acid on perhydrolysis. Suitable O-acylated perbenzoic acid precursor compounds include the substituted and unsubstituted benzoyl oxybenzene sulfonates, and the benzoylation products of sorbitol, glucose, and all saccharides with benzoylating agents, and those of the imide type including N-benzoyl succinimide, tetrabenzoyl ethylene diamine and the N-benzoyl substituted ureas. Suitable imidazole type perbenzoic acid precursors include N-benzoyl imidazole and N-benzoyl benzimidazole. Other useful N-acyl group-containing perbenzoic acid precursors include N-benzoyl pyrrolidone, dibenzoyl taurine and benzoyl pyroglutamic acid.

### Cationic peroxyacid precursors

Cationic peroxyacid precursor compounds produce cationic peroxyacids on perhydrolysis.

Typically, cationic peroxyacid precursors are formed by substituting the peroxyacid part of a suitable peroxyacid precursor compound with a positively charged functional group, such as an ammonium or alkyl ammonium group, preferably an ethyl or methyl ammonium group. Cationic peroxyacid precursors are typically present in the solid detergent compositions as a salt with a suitable anion, such as a halide ion.

The peroxyacid precursor compound to be so cationically substituted may be a perbenzoic acid, or substituted derivative thereof, precursor compound as described hereinbefore. Alternatively, the peroxyacid precursor compound may be an alkyl percarboxylic acid precursor compound or an amide substituted alkyl peroxyacid precursor as described hereinafter.



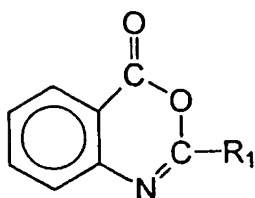
Cationic peroxyacid precursors are described in U.S. Patents 4,904,406; 4,751,015; 4,988,451; 4,397,757; 5,269,962; 5,127,852; 5,093,022; 5,106,528; U.K. 1,382,594; EP 475,512, 458,396 and 284,292; and in JP 87-318,332.

Examples of preferred cationic peroxyacid precursors are described in UK Patent Application No. 9407944.9 and US Patent Application Nos. 08/298903, 08/298650, 08/298904 and 08/298906.

Suitable cationic peroxyacid precursors include any of the ammonium or alkyl ammonium substituted alkyl or benzoyl oxybenzene sulfonates, N-acylated caprolactams, and monobenzoyltetraacetyl glucose benzoyl peroxides. Preferred cationic peroxyacid precursors of the N-acylated caprolactam class include the trialkyl ammonium methylene benzoyl caprolactams and the trialkyl ammonium methylene alkyl caprolactams.

#### Benzoxazin organic peroxyacid precursors

Also suitable are precursor compounds of the benzoxazin-type, as disclosed for example in EP-A-332,294 and EP-A-482,807, particularly those having the formula:

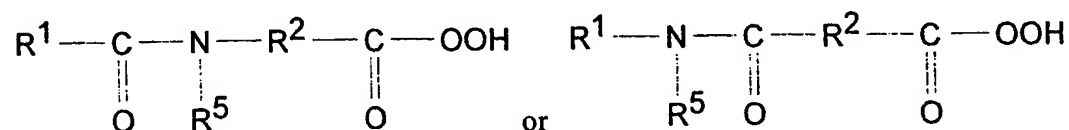


wherein  $R_1$  is H, alkyl, alkaryl, aryl, or arylalkyl.

#### Preformed organic peroxyacid

The organic peroxyacid bleaching system may contain, in addition to, or as an alternative to, an organic peroxyacid bleach precursor compound, a preformed organic peroxyacid, typically at a level of from 1% to 15% by weight, more preferably from 1% to 10% by weight of the composition.

A preferred class of organic peroxyacid compounds are the amide substituted compounds of the following general formulae:



wherein  $\text{R}^1$  is an alkyl, aryl or alkaryl group with from 1 to 14 carbon atoms,  $\text{R}^2$  is an alkylene, arylene, and alkarylene group containing from 1 to 14 carbon atoms, and  $\text{R}^5$  is H or an alkyl, aryl, or alkaryl group containing 1 to 10 carbon atoms. Amide substituted organic peroxyacid compounds of this type are described in EP-A-0170386.

Other organic peroxyacids include diacyl and tetraacylperoxides, especially diperoxydodecanedioic acid, diperoxytetradecanedioic acid and diperoxyhexadecanedioic acid. Mono- and diperazelaic acid, mono- and diperbrassylic acid and N-phthaloylaminoperoxicaproic acid are also suitable herein.

#### Heavy metal ion sequestrant

The particle or compositions herein preferably contain as an optional component a heavy metal ion sequestrant. By heavy metal ion sequestrant it is meant herein components which act to sequester (chelate) heavy metal ions. These components may also have calcium and magnesium chelation capacity, but preferentially they show selectivity to binding heavy metal ions such as iron, manganese and copper.

Heavy metal ion sequestrants are generally present at a level of from 0.005% to 10%, preferably from 0.1% to 5%, more preferably from 0.25% to 7.5% and most preferably from 0.3% to 2% by weight of the compositions or component

Suitable heavy metal ion sequestrants for use herein include organic phosphonates, such as the amino alkylene poly (alkylene phosphonates), alkali metal ethane 1-hydroxy diphosphonates and nitrilo trimethylene phosphonates.

Preferred among the above species are diethylene triamine penta (methylene phosphonate), ethylene diamine tri (methylene phosphonate) hexamethylene diamine tetra (methylene phosphonate) and hydroxy-ethylene 1,1 diphosphonate and 1,1 hydroxyethane diphosphonic acid.

Other suitable heavy metal ion sequestrant for use herein include nitrilotriacetic acid and polyaminocarboxylic acids such as ethylenediaminetetracetic acid, ethylenediamine disuccinic acid, ethylenediamine diglutaric acid, 2-hydroxypropylenediamine disuccinic acid or any salts thereof.

Other suitable heavy metal ion sequestrants for use herein are iminodiacetic acid derivatives such as 2-hydroxyethyl diacetic acid or glyceryl imino diacetic acid, described in EP-A-317,542 and EP-A-399,133. The iminodiacetic acid-N-2-hydroxypropyl sulfonic acid and aspartic acid N-carboxymethyl N-2-hydroxypropyl-3-sulfonic acid sequestrants described in EP-A-516,102 are also suitable herein. The  $\beta$ -alanine-N,N'-diacetic acid, aspartic acid-N,N'-diacetic acid, aspartic acid-N-monoacetic acid and iminodisuccinic acid sequestrants described in EP-A-509,382 are also suitable.

EP-A-476,257 describes suitable amino based sequestrants. EP-A-510,331 describes suitable sequestrants derived from collagen, keratin or casein. EP-A-528,859 describes a suitable alkyl iminodiacetic acid sequestrant. Dipicolinic acid and 2-phosphonobutane-1,2,4-tricarboxylic acid are also suitable. Glycinamide-

N,N'-disuccinic acid (GADS), ethylenediamine-N-N'-diglutamic acid (EDDG) and 2-hydroxypropylenediamine-N-N'-disuccinic acid (HPDDS) are also suitable.

Especially preferred are diethylenetriamine pentacetic acid, ethylenediamine-N,N'-disuccinic acid (EDDS) and 1,1 hydroxyethane diphosphonic acid or the alkali metal, alkaline earth metal, ammonium, or substituted ammonium salts thereof, or mixtures thereof.

### Enzyme

Another preferred ingredient useful in the compositions is one or more additional enzymes.

Preferred additional enzymatic materials include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139.

Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Protease enzyme may be incorporated into the compositions in accordance with the invention at a level of from 0.0001% to 4% active enzyme by weight of the composition.

Preferred amylases include, for example,  $\alpha$ -amylases obtained from a special strain of *B licheniformis*, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyland BAN by

Novo Industries A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635.

Amylase enzyme may be incorporated into the composition in accordance with the invention at a level of from 0.0001% to 2% active enzyme by weight of the composition.

Lipolytic enzyme may be present at levels of active lipolytic enzyme of from 0.0001% to 2% by weight, preferably 0.001% to 1% by weight, most preferably from 0.001% to 0.5% by weight of the compositions.

The lipase may be fungal or bacterial in origin being obtained, for example, from a lipase producing strain of Humicola sp., Thermomyces sp. or Pseudomonas sp. including Pseudomonas pseudoalcaligenes or Pseudomas fluorescens. Lipase from chemically or genetically modified mutants of these strains are also useful herein. A preferred lipase is derived from Pseudomonas pseudoalcaligenes, which is described in Granted European Patent, EP-B-0218272.

Another preferred lipase herein is obtained by cloning the gene from Humicola lanuginosa and expressing the gene in Aspergillus oryza, as host, as described in European Patent Application, EP-A-0258 068, which is commercially available from Novo Industri A/S, Bagsvaerd, Denmark, under the trade name Lipolase. This lipase is also described in U.S. Patent 4,810,414, Høge-Jensen et al, issued March 7, 1989.

#### Organic Polymeric Compound

Organic polymeric compounds are preferred additional components of the compositions or particles herein and are preferably present as components of any particulate components where they may act such as to bind the particulate component together. By organic polymeric compound it is meant herein essentially any polymeric organic compound commonly used as dispersants, and anti-redeposition and soil suspension

agents in detergent compositions, including any of the high molecular weight organic polymeric compounds described as clay flocculating agents herein, not being an quaternised ethoxylated (poly) amine clay-soil removal/ anti-redeposition agent in accord with the invention.

Organic polymeric compound is typically incorporated in the detergent compositions of the invention at a level of from 0.01% to 30%, preferably from 0.1% to 15%, most preferably from 0.5% to 10% by weight of the compositions.

Examples of organic polymeric compounds include the water soluble organic homo- or co-polymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxyl radicals separated from each other by not more than two carbon atoms. Polymers of the latter type are disclosed in GB-A-1,596,756. Examples of such salts are polyacrylates of MWt 1000-5000 and their copolymers with maleic anhydride, such copolymers having a molecular weight of from 2000 to 100,000, especially 40,000 to 80,000.

The polyamino compounds are useful herein including those derived from aspartic acid such as those disclosed in EP-A-305282, EP-A-305283 and EP-A-351629.

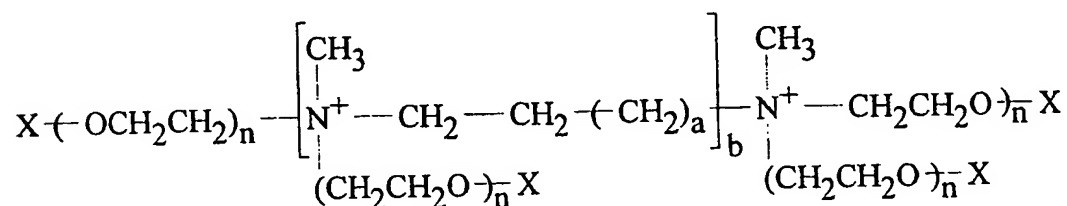
Terpolymers containing monomer units selected from maleic acid, acrylic acid, polyaspartic acid and vinyl alcohol, particularly those having an average molecular weight of from 5,000 to 10,000, are also suitable herein.

Other organic polymeric compounds suitable for incorporation in the detergent compositions herein include cellulose derivatives such as methylcellulose, carboxymethylcellulose, hydroxypropylmethylcellulose and hydroxyethylcellulose.

Further useful organic polymeric compounds are the polyethylene glycols, particularly those of molecular weight 1000-10000, more particularly 2000 to 8000 and most preferably about 4000.

Highly preferred polymeric components herein are cotton and non-cotton soil release polymer according to U.S. Patent 4,968,451, Scheibel et al., and U.S. Patent 5,415,807, Gosselink et al., and in particular according to US application no.60/051517.

Another organic compound, which is a preferred clay dispersant/ anti-redeposition agent, for use herein, can be the ethoxylated cationic monoamines and diamines of the formula:



wherein X is a nonionic group selected from the group consisting of H, C<sub>1</sub>-C<sub>4</sub> alkyl or hydroxyalkyl ester or ether groups, and mixtures thereof, a is from 0 to 20, preferably from 0 to 4 (e.g. ethylene, propylene, hexamethylene) b is 1 or 0; for cationic monoamines (b=0), n is at least 16, with a typical range of from 20 to 35; for cationic diamines (b=1), n is at least about 12 with a typical range of from about 12 to about 42.

Other dispersants/ anti-redeposition agents for use herein are described in EP-B-011965 and US 4,659,802 and US 4,664,848.

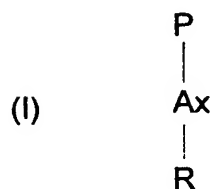
#### Polymeric dye transfer inhibiting agents

The particle or the compositions herein may also comprise from 0.01% to 10 %, preferably from 0.05% to 0.5% by weight of polymeric dye transfer inhibiting agents.

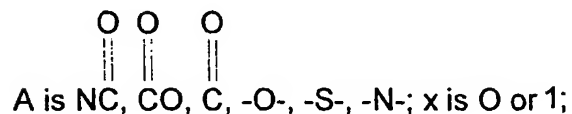
The polymeric dye transfer inhibiting agents are preferably selected from polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidone polymers or combinations thereof.

a) Polyamine N-oxide polymers

Polyamine N-oxide polymers suitable for use herein contain units having the following structure formula :



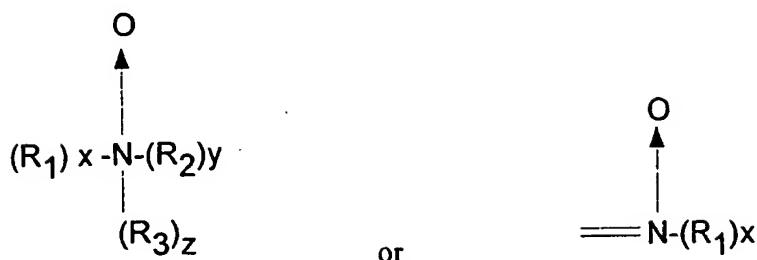
wherein P is a polymerisable unit, and



R are aliphatic, ethoxylated aliphatics, aromatic, heterocyclic or alicyclic groups or any combination thereof whereto the nitrogen of the N-O group can be attached or wherein the nitrogen of the N-O group is part of these groups.

The N-O group can be represented by the following general structures :





wherein R1, R2, and R3 are aliphatic groups, aromatic, heterocyclic or alicyclic groups or combinations thereof, x or/and y or/and z is 0 or 1 and wherein the nitrogen of the N-O group can be attached or wherein the nitrogen of the N-O group forms part of these groups. The N-O group can be part of the polymerisable unit (P) or can be attached to the polymeric backbone or a combination of both.

Suitable polyamine N-oxides wherein the N-O group forms part of the polymerisable unit comprise polyamine N-oxides wherein R is selected from aliphatic, aromatic, alicyclic or heterocyclic groups. One class of said polyamine N-oxides comprises the group of polyamine N-oxides wherein the nitrogen of the N-O group forms part of the R-group. Preferred polyamine N-oxides are those wherein R is a heterocyclic group such as pyridine, pyrrole, imidazole, pyrrolidine, piperidine, quinoline, acridine and derivatives thereof.

Other suitable polyamine N-oxides are the polyamine oxides whereto the N-O group is attached to the polymerisable unit. A preferred class of these polyamine N-oxides comprises the polyamine N-oxides having the general formula (I) wherein R is an aromatic, heterocyclic or alicyclic groups wherein the nitrogen of the N-O functional group is part of said R group. Examples of these classes are polyamine oxides wherein R is a heterocyclic compound such as pyridine, pyrrole, imidazole and derivatives thereof.

The polyamine N-oxides can be obtained in almost any degree of polymerisation. The degree of polymerisation is not critical provided the material has the desired water-

solubility and dye-suspending power. Typically, the average molecular weight is within the range of 500 to 1000,000.

b) Copolymers of N-vinylpyrrolidone and N-vinylimidazole

Suitable herein are copolymers of N-vinylimidazole and N-vinylpyrrolidone having an average molecular weight range of from 5,000 to 50,000. The preferred copolymers have a molar ratio of N-vinylimidazole to N-vinylpyrrolidone from 1 to 0.2.

c) Polyvinylpyrrolidone

The components or the compositions herein may also utilize polyvinylpyrrolidone ("PVP") having an average molecular weight of from 2,500 to 400,000. Suitable polyvinylpyrrolidones are commercially available from ISP Corporation, New York, NY and Montreal, Canada under the product names PVP K-15 (viscosity molecular weight of 10,000), PVP K-30 (average molecular weight of 40,000), PVP K-60 (average molecular weight of 160,000), and PVP K-90 (average molecular weight of 360,000). PVP K-15 is also available from ISP Corporation. Other suitable polyvinylpyrrolidones which are commercially available from BASF Cooperation include Sokalan HP 165 and Sokalan HP 12.

d) Polyvinylloxazolidone

The compositions herein may also utilize polyvinylloxazolidones as polymeric dye transfer inhibiting agents. Said polyvinylloxazolidones have an average molecular weight of from 2,500 to 400,000.

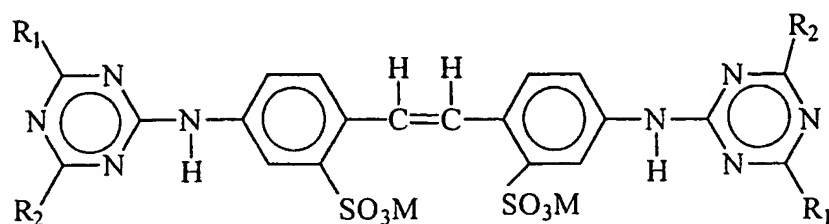
e) Polyvinylimidazole

The compositions herein may also utilize polyvinylimidazole as polymeric dye transfer inhibiting agent. Said polyvinylimidazoles preferably have an average molecular weight of from 2,500 to 400,000.

### Optical brightener

The compositions or particles herein also optionally contain from about 0.005% to 5% by weight of certain types of hydrophilic optical brighteners.

Hydrophilic optical brighteners useful herein include those having the structural formula:



wherein  $R_1$  is selected from anilino, N-2-bis-hydroxyethyl and NH-2-hydroxyethyl;  $R_2$  is selected from N-2-bis-hydroxyethyl, N-2-hydroxyethyl-N-methylamino, morphilino, chloro and amino; and M is a salt-forming cation such as sodium or potassium.

When in the above formula,  $R_1$  is anilino,  $R_2$  is N-2-bis-hydroxyethyl and M is a cation such as sodium, the brightener is 4,4',-bis[(4-anilino-6-(N-2-bis-hydroxyethyl)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid and disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal-UNPA-GX by Ciba-Geigy Corporation. Tinopal-UNPA-GX is the preferred hydrophilic optical brightener useful in the detergent compositions herein.

When in the above formula,  $R_1$  is anilino,  $R_2$  is N-2-hydroxyethyl-N-2-methylamino and M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-(N-2-hydroxyethyl-

N-methylamino)-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid disodium salt. This particular brightener species is commercially marketed under the tradename Tinopal 5BM-GX by Ciba-Geigy Corporation.

When in the above formula, R<sub>1</sub> is anilino, R<sub>2</sub> is morphilino and M is a cation such as sodium, the brightener is 4,4'-bis[(4-anilino-6-morphilino-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid, sodium salt. This particular brightener species is commercially marketed under the tradename Tinopal AMS-GX by Ciba Geigy Corporation.

#### Cationic fabric softening agents

Cationic fabric softening agents can also be incorporated into the compositions or particles herein. Suitable cationic fabric softening agents include the water insoluble tertiary amines or dilong chain amide materials as disclosed in GB-A-1 514 276 and EP-B-0 011 340.

Cationic fabric softening agents are typically incorporated at total levels of from 0.5% to 15% by weight, normally from 1% to 5% by weight.

#### Other optional ingredients

Other optional ingredients suitable for inclusion in the particle of the invention or the compositions herein of include highly preferably perfumes, bleach catalysts, colours and filler salts, with sodium sulfate being a preferred filler salt.

#### pH of the compositions

The present particle or the cleaning compositions can have an acidic, neutral or an alkaline pH, depending on the application or the additional ingredients comprised in the component or composition. Preferably, the component or the compositions herein have a

pH measured as a 1% solution in distilled water, of from 3 to 13.5, preferably least 4.0, preferably from to 12.5, most preferably from 5 to 12.0.

#### Form of the cleaning compositions

The cleaning compositions, comprising the component, can be made via a variety of methods, including dry-mixing, extruding, compressing and agglomerating of the various components comprised in the detergent composition.

The cleaning compositions can take a variety of physical forms including non-aqueous, and solid compositions, including granular, extrudates tablet or bar forms. The cleaning compositions are particularly the so-called concentrated granular detergent compositions adapted to be added to a washing or dish washing machine by means of a dispensing drawer or by means of a dispensing device placed in the machine drum with the soiled fabric load.

The mean particle size of the base composition of granular cleaning compositions containing the foaming composition in accordance with the invention can be from 0.1 mm to 5.0 mm, but it should preferably be such that no more than 5% of particles are greater than 2.5mm in diameter, or even 1.7mm and that not more than 5% of particles are less than 0.15mm in diameter.

The term mean particle size as defined herein is calculated by sieving a sample of the composition into a number of fractions (typically 5 fractions) on a series of Tyler sieves. The weight fractions thereby obtained are plotted against the aperture size of the sieves. The mean particle size is taken to be the aperture size through which 50% by weight of the sample would pass.

The bulk density of granular cleaning or detergent compositions containing the particulate composition in accordance with the present invention typically have a bulk

density of at least 300 g/litre, more preferably from 500 g/litre or even 650g/litre to 1200 g/litre, more preferably to 850 g/litre. Bulk density is measured by means of a simple funnel and cup device consisting of a conical funnel moulded rigidly on a base and provided with a flap valve at its lower extremity to allow the contents of the funnel to be emptied into an axially aligned cylindrical cup disposed below the funnel. The funnel is 130 mm high and has internal diameters of 130 mm and 40 mm at its respective upper and lower extremities. It is mounted so that the lower extremity is 140 mm above the upper surface of the base. The cup has an overall height of 90 mm, an internal height of 87 mm and an internal diameter of 84 mm. Its nominal volume is 500 ml.

To carry out a measurement, the funnel is filled with powder by hand pouring, the flap valve is opened and powder allowed to overfill the cup. The filled cup is removed from the frame and excess powder removed from the cup by passing a straight edged implement eg; a knife, across its upper edge. The filled cup is then weighed and the value obtained for the weight of powder doubled to provide a bulk density in g/litre. Replicate measurements are made as required.

The composition is preferably soluble in cold or cool water, i.e. the composition readily dissolves/disperses in water at a temperature between about 0°C and 32.2°C, preferably between about 1.6°C and 10°C.

#### Laundry washing method

Machine laundry methods herein typically comprise treating soiled laundry with an aqueous wash solution in a washing machine having dissolved or dispensed therein an effective amount of a machine laundry particle of the invention or composition comprising the foaming component of the invention. By an effective amount of the detergent composition it is meant from 40g to 300g of product dissolved or dispersed in a

wash solution of volume from 5 to 65 litres, as are typical product dosages and wash solution volumes commonly employed in conventional machine laundry methods.

### Abbreviations used in Examples

In the exemplified particles and compositions, the abbreviated component identifications have the following meanings:

|                                  |   |  |
|----------------------------------|---|--|
| LAS                              | : | Sodium linear C <sub>12</sub> alkyl benzene sulfonate  |
| TAS                              | : | Sodium tallow alkyl sulfate  |
| C45AS                            | : | Sodium C <sub>14</sub> -C <sub>15</sub> linear alkyl sulfate   |
| MES                              | : | $\alpha$ -sulpho methylester of C <sub>18</sub> fatty acid   |
| C <sub>x</sub> yE <sub>z</sub> S | : | Sodium C <sub>1x</sub> -C <sub>1y</sub> branched alkyl sulfate condensed<br>with z moles of ethylene oxide   |
| MBAS <sub>x, y</sub>             | : | Sodium mid-chain branched alkyl sulfate<br>having an average of x carbon atoms, whereof an<br>average of y carbons comprised in (a)<br>branching unit(s)   |
| C <sub>48</sub> SAS              | : | Sodium C <sub>14</sub> -C <sub>18</sub> secondary alcohol sulfate  |
| SADE <sub>x</sub> S              | : | Sodium C <sub>14</sub> -C <sub>22</sub> alkyl disulfate of formula 2-(R).C <sub>4</sub> H <sub>7</sub> -1,4-<br>(SO <sub>4</sub> ) <sub>2</sub> where R = C <sub>10</sub> 0C <sub>18</sub> , condensed with z moles of<br>ethylene oxide |
| C <sub>x</sub> yE <sub>z</sub>   | : | A C <sub>1x-1y</sub> branched primary alcohol condensed with an<br>average of z moles of ethylene oxide  |
| QAS I                            | : | R <sub>2</sub> .N <sup>+</sup> (CH <sub>3</sub> ) <sub>2</sub> (C <sub>2</sub> H <sub>4</sub> OH) with R <sub>2</sub> = 50%-60% C <sub>9</sub> ; 40%-<br>50% C <sub>11</sub>   |
| QAS II                           | : | R <sub>1</sub> .N <sup>+</sup> (CH <sub>3</sub> )(C <sub>2</sub> H <sub>4</sub> OH) <sub>2</sub> with R <sub>1</sub> = C <sub>12</sub> -C <sub>14</sub>  |
| Soap                             | : | Sodium linear alkyl carboxylate derived from an 80/20<br>mixture of tallow and coconut oils.   |

|                |   |  |
|----------------|---|--|
| TFAA I         | : | C <sub>12</sub> -C <sub>14</sub> alkyl N-methyl glucamide  |
| TFAA II        | : | C <sub>16</sub> -C <sub>18</sub> alkyl N-methyl glucamide  |
| TPKFA          | : | C <sub>12</sub> -C <sub>14</sub> topped whole cut fatty acids  |
| STPP           | : | Anhydrous sodium tripolyphosphate  |
| Zeolite A I    | : | Hydrated Sodium Aluminosilicate of formula<br>Na <sub>12</sub> (Al <sub>10</sub> Si <sub>2</sub> O <sub>2</sub> ) <sub>12</sub> · 27H <sub>2</sub> O having a primary particle size<br>in the range from 0.1 to 10 micrometers |
| Zeolite A II   | : | overdried Zeolite A I  |
| NaSKS-6        | : | Crystalline layered silicate of formula $\delta$ -Na <sub>2</sub> Si <sub>2</sub> O <sub>5</sub>   |
| Citric acid I  | : | Anhydrous citric acid  |
| Citric acid II | : | Citric acid monohydrate  |
| Malic acid     | : | Anhydrous malic acid   |
| Maleic acid    | : | Anhydrous maleic acid  |
| Aspartic acid  | : | Anhydrous aspartic acid  |
| Carbonate I    | : | Anhydrous sodium carbonate with an average particle size<br>between 200 $\mu$ m and 900 $\mu$ m  |
| Carbonate II   | : | Anhydrous sodium carbonate with an average particle size<br>between 100 $\mu$ m and 200 $\mu$ m  |
| Bicarbonate    | : | Anhydrous sodium bicarbonate with a particle size<br>distribution between 400 $\mu$ m and 1200 $\mu$ m   |
| Silicate       | : | Amorphous Sodium Silicate (SiO <sub>2</sub> :Na <sub>2</sub> O; 2.0 ratio)   |
| Sodium sulfate | : | Anhydrous sodium sulfate   |
| Citrate        | : | Tri-sodium citrate dihydrate of activity 86.4% with a<br>particle size distribution between 425 $\mu$ m and q 850 $\mu$ m  |
| MA/AA          | : | Copolymer of 1:4 maleic/acrylic acid, average molecular<br>weight about 70,000   |
| CMC            | : | Sodium carboxymethyl cellulose   |
| Protease       | : | Proteolytic enzyme of activity 4KNPU/g sold by NOVO<br>Industries A/S under the tradename Savinase   |



|                |   |   |
|----------------|---|---|
| Alcalase       | : | Proteolytic enzyme of activity 3AU/g sold by NOVO Industries A/S  |
| Cellulase      | : | Cellulytic enzyme of activity 1000 CEVU/g sold by NOVO Industries A/S under the tradename Carezyme                    |
| Amylase        | : | Amylolytic enzyme of activity 60KNU/g sold by NOVO Industries A/S under the tradename Termamyl 60T                    |
| Lipase         | : | Lipolytic enzyme of activity 100kLU/g sold by NOVO Industries A/S under the tradename Lipolase                        |
| Endolase       | : | Endoglunase enzyme of activity 3000 CEVU/g sold by NOVO Industries A/S  |
| PB4            | : | Sodium perborate tetrahydrate of nominal formula $\text{NaBO}_2 \cdot 3\text{H}_2\text{O} \cdot \text{H}_2\text{O}_2$ |
| PB1            | : | Anhydrous sodium perborate bleach of nominal formula $\text{NaBO}_2 \cdot \text{H}_2\text{O}_2$                       |
| Percarbonate   | : | Sodium Percarbonate of nominal formula $2\text{Na}_2\text{CO}_3 \cdot 3\text{H}_2\text{O}_2$                          |
| NAC-OBS        | : | (Nonanamido caproyl) oxybenzene sulfonate in the form of the sodium salt.   |
| NOBS           | : | Nonanoyl oxybenzene sulfonate in the form of the sodium salt  |
| DPDA           | : | Diperoxydodecanedioic acid  |
| PAP            | : | N-phthaloylamidoperoxicaproic acid  |
| NAPAA          | : | Nonanoylamido peroxo-adipic acid  |
| NACA           | : | 6 nonylamino - 6 oxo - capronic acid.   |
| TAED           | : | Tetraacetythylenediamine  |
| DTPMP          | : | Diethylene triamine penta (methylene phosphonate), marketed by Monsanto under the Trade name Dequest 2060             |
| Photoactivated | : | Sulfonated Zinc or aluminium Phthlocyanine encapsulated   |
| Brightener 1   | : | Disodium 4,4'-bis(2-sulphostyryl)biphenyl   |

|                   |   |  |
|-------------------|---|--|
| Brightener 2      | : | Disodium 4,4'-bis(4-anilino-6-morpholino-1.3.5-triazin-2-yl)amino) stilbene-2:2'-disulfonate.  |
| HEDP              | : | 1,1-hydroxyethane diphosphonic acid  |
| PVNO              | : | Polyvinylpyridine N-oxide  |
| PVPVI             | : | Copolymer of polyvinylpyrrolidone and vinylimidazole   |
| QEA               | : | bis ((C <sub>2</sub> H <sub>5</sub> O)(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub> ) (CH <sub>3</sub> ) -N <sup>+</sup> -C <sub>6</sub> H <sub>12</sub> -N <sup>+</sup> - (CH <sub>3</sub> )<br>bis ((C <sub>2</sub> H <sub>5</sub> O)-(C <sub>2</sub> H <sub>4</sub> O) <sub>n</sub> ), wherein n=from 20 to 30 |
| SRP 1             | : | Sulfo benzoyl end capped esters with oxyethylene oxy and terephthaloyl backbone  |
| SRP 2             | : | Diethoxylated poly (1, 2 propylene terephthalate) short block polymer  |
| Silicone antifoam | : | Polydimethylsiloxane foam controller with siloxane-oxyalkylene copolymer as dispersing agent with a ratio of said foam controller to said dispersing agent of 10:1 to 100:1.   |

In the following Examples all levels are quoted as parts per weight of the composition or % by weight of the composition, as indicated:

### Particles A to H

The following are examples of particles in accord with the invention:

|                            | A  | B  | C  | D  | E  | F  | G  | H  |
|----------------------------|----|----|----|----|----|----|----|----|
| Anhydrous sodium perborate | 30 | 50 | 60 | 45 | 55 | 50 | 50 | 40 |
| sodium sulphate            | 5  | -  | -  | -  | 5  |    | -  | -  |
| citric acid /malic acid    | -  | 10 | 10 | -  | 10 | -  | -  | -  |

|                     |    |    |    |    |    |    |    |      |
|---------------------|----|----|----|----|----|----|----|------|
| sodium carbonate    | 15 | 10 | 20 | -  | 25 | 10 | -  | 10   |
| potassium carbonate | 5  | 10 | 5  | -  | -  | -  | -  | -    |
| Zeolite A           | 10 | 10 | -  | 10 | -  | -  | -  | -    |
| C24E3/C24E5         | -  | -  | -  | 20 | -  | -  | -  | 10.0 |
| sodium SKS-6        | -  | -  | 5  | -  | -  | -  | 5  | -    |
| sodium chloride     | 5  | -  | 5  | -  | -  | -  | -  | -    |
| MA/AA               | 10 | -  | -  | -  | -  | 20 | -  | -    |
| CFAA                | -  | -  | 20 | -  | -  | -  | 15 | 20   |
| LAS                 | 10 | 10 | -  | -  | -  | -  | 17 | -    |
| QAS I/ QAS II       | -  | -  | -  | 5  | -  | 5  | -  | -    |
| TAED/NOBS/NACA-OBS  | 5  | -  | -  | -  | -  | -  | -  | 5    |
| sodium percarbonate | 10 | -  | -  | -  | -  | 17 | -  | 10   |
| silicon antifoam    | -  | -  | -  | -  | -  | -  | 10 | -    |

**Coating:**

|                |   |   |   |   |   |   |   |   |
|----------------|---|---|---|---|---|---|---|---|
| MA/AA, MW 4000 | - | - | - | - | 5 | - | - | - |
| C24 fatty acid | - | - | - | - | 3 | - | - | - |
| wax            | - | - | - | - | - | - | 3 | - |
| paraffin oil   | - | - | - | - | - | - | - | 5 |

The following examples exemplify cleaning compositions comprising the foaming component of the invention:

**Example I**

The following are high density and bleach-containing detergent formulations according to the present invention:

|                | a   | b   | c    |
|----------------|-----|-----|------|
| Blown Powder   |     |     |      |
| Zeolite A      | 5.0 | 5.0 | 15.0 |
| Sodium sulfate | 0.0 | 5.0 | 0.0  |
| LAS            | -   | 5.0 | 3.0  |
| C45AS          | 3.0 | 2.0 | 4.0  |

|                                    |      |      |      |
|------------------------------------|------|------|------|
| QAS                                | -    | -    | 1.5  |
| DTPMP                              | 0.4  | 0.4  | 0.4  |
| CMC                                | 0.4  | 0.4  | 0.4  |
| MA/AA                              | 4.0  | 2.0  | 2.0  |
| Spray On (on particles)            |      |      |      |
| Encapsulated Perfume               | 0.3  | 0.3  | 0.3  |
| C25E3                              | -    | -    | 2.0  |
| Dry additives                      |      |      |      |
| QEA                                | -    | -    | 0.5  |
| Citrate                            | 5.0  | -    | 2.0  |
| Bicarbonate                        | -    | 3.0  | -    |
| Carbonate                          | 8.0  | 10.0 | 5.0  |
| NAC OBS                            | 6.0  | -    | -    |
| Manganese catalyst                 | -    | -    | 0.3  |
| NOBS                               | -    | 2.0  | -    |
| PB1                                | 14.0 | 7.0  | -    |
| Polyethylene oxide of MW 5,000,000 | -    | -    | 0.2  |
| Bentonite clay                     | -    | -    | 10.0 |
| Citric acid                        | -    | -    | 0.5  |
| Protease                           | 1.0  | 1.0  | 1.0  |
| Lipase                             | 0.4  | 0.4  | 0.4  |
| Amylase                            | 0.6  | 0.6  | 0.6  |
| Cellulase                          | 0.6  | 0.6  | 0.6  |
| Silicone antifoam                  | 5.0  | 5.0  | 5.0  |
| Particle A/D                       | 20.0 |      |      |
| Particle B/H                       | -    | 15.0 | -    |
| Particle E                         | -    | -    | 10.0 |
|                                    |      |      |      |

|                                      |       |       |       |
|--------------------------------------|-------|-------|-------|
| Balance (Moisture and Miscellaneous) | 100.0 | 100.0 | 100.0 |
| Density (g/litre)                    | 580   | 650   | 850   |

**Example 2**

The following are detergent formulations according to the present invention:

|              | d    | e    |
|--------------|------|------|
| Particle B/C | 20.0 |      |
| Particle F/H |      | 30.0 |
| C25E3        | -    | 1.0  |
| Perfume      | 0.5  | 0.5  |
|              |      |      |
| HEDP         | 0.5  | 0.3  |
| SKS 6        | 13.0 | 10.0 |
| Citrate      | -    | 1.0  |
| NAC OBS      | 4.1  | -    |
| TAED         | 0.8  | -    |
| Percarbonate | 20.0 | 5.0  |
| SRP 1        | 0.3  | 0.3  |
| Protease     | 1.4  | 1.4  |
| Lipase       | 0.4  | 0.4  |
| Cellulase    | 0.6  | 0.6  |
| Amylase      | 0.6  | 0.6  |
| QEA          | 1.0  | -    |

|                   |     |     |
|-------------------|-----|-----|
| Silicone antifoam | 5.0 | 5.0 |
| Brightener 1      | 0.2 | 0.2 |
| Brightener 2      | 0.2 | -   |
| Density (g/litre) | 850 | 850 |

**Example 3**

The following granular detergent formulations are examples of the present invention.

|                | f    | g    | h    |
|----------------|------|------|------|
| Blown powder   |      |      |      |
| MES            | -    | 2.0  | -    |
| LAS            | 2.0  | -    | -    |
| C45AS          | -    | 1.0  | -    |
| C46AS          | 4.0  | -    | -    |
| C45AE35        | 2.0  | 5.0  | 3.0  |
| MBAS18, 4.0    | 8.0  | -    | -    |
| Zeolite A      | 16.0 | 19.0 | 6.0  |
| MA/AA          | 3.0  | -    | -    |
| AA             | 3.0  | 2.0  | 3.0  |
| Sodium sulfate | 3.3  | 24.0 | 13.3 |
| Silicate       | 1.0  | 2.0  | 1.0  |
| Carbonate      | 9.0  | 25.7 | 8.0  |
| QEA            | 0.4  | -    | 0.5  |
| PEG 4000       | -    | 1.0  | 1.5  |
| Brightener     | 0.3  | 0.3  | 0.3  |
| Spray on       |      |      |      |
| C25E5          | 0.5  | 1.0  | -    |

| Perfume              | 0.3  | 1.0  | 0.3  |
|----------------------|------|------|------|
| dry added:           |      |      |      |
| Particle A, E or H   | -    | -    | 20.0 |
| Particle D or F or I | -    | 15.0 | -    |
| Particle B or C      | 10.0 | -    | -    |
| NACAOBS/NOBS         | 3.0  | -    | -    |
| TAED                 | 1.0  | -    | 2.0  |
| PB4                  | 1.0  | -    | 2.0  |
| Carbonate            | 5.3  | -    | 2.5  |
| Cumene sulfonic acid | 2.0  | -    | 2.0  |
| Lipase               | 0.4  | 0.1  | 0.05 |
| Cellulase            | 0.2  | -    | 0.2  |
| Amylase              | 0.3  | -    | -    |
| Protease             | 1.6  | -    | 1.6  |
| PVPVI                | 0.5  | -    | -    |
| PVNO                 | 0.5  | -    | -    |
| SRP1                 | 0.5  | -    | -    |
| Silicone antifoam    | 1.0  | -    | 0.2  |

**Example 4**

The following tablet formulation comprises the particle of the invention:

|      | i    | j    | k    | l    | m    | n    |
|------|------|------|------|------|------|------|
| LAS  | 20.0 | 15.0 | 25.0 | 30.0 | 10.0 | 18.0 |
| AS   | 3.0  | 0.5  | -    | -    | 7.0  | 3.0  |
| TFAA | 2.0  | 2.5  | 3.5  | -    | -    | -    |



|                       |      |      |      |      |      |      |
|-----------------------|------|------|------|------|------|------|
| C25E3                 | -    | -    | -    | 1.0  | 4.0  | 2.0  |
| C25E7                 | 3.0  | 3.0  | -    | -    | -    | -    |
| QAS                   | 0.5  | 0.8  | 2.0  | -    | -    | 1.0  |
| Particle A/D/G        | -    | -    | -    | 5.0  | 20.0 | -    |
| Particle B/ E/ F      | -    | -    | 10.0 | -    | -    | 7.0  |
| Particle C/H          | 3.0  | 15.0 | -    | -    | -    | -    |
| Zeolite A             | 10.0 | 10.0 | 15.0 | 5.0  | 6.0  | 10.0 |
| Citric acid           | -    | -    | -    | 25.0 | -    | 2.5  |
| MA/AA                 | 1.0  | 1.0  | 3.0  | -    | -    | 0.5  |
| SKS-6                 | 5.0  | 3.0  | 2.0  | -    | -    | 10.0 |
| Silicate              | 1.0  | 1.0  | 3.0  | 0.3  | -    | -    |
| carbonate/bicarbonate | 5.0  | -    | 10.0 | 3.0  | -    | 12.0 |
| Sulfate               | 10.0 | -    | 5.0  | -    | 6.0  | -    |
| Mg sulfate            | 0.3  | -    | -    | 0.2  | -    | 0.2  |
| QEA                   | 0.3  | 0.3  | 0.3  | 4.0  | 1.0  | 1.0  |
| CMC                   | 0.2  | -    | 0.2  | 0.2  | 0.4  | 0.4  |

|                                |        |        |      |      |        |        |
|--------------------------------|--------|--------|------|------|--------|--------|
| SRP1                           | 1.0    | 0.5    | -    | -    | 1.0    | -      |
| Percarbonate                   | 10.0   | -      | 15.0 | -    | 10.0   | -      |
| TAED                           | 1.5    | -      | 1.5  | -    | 4.0    | -      |
| NAC-OBS/NOBS                   | 3.0    | -      | 5.0  | -    | -      | -      |
| DTPMP                          | -      | -      | 0.25 | 0.25 | -      | -      |
| HEDP                           | 0.5    | -      | -    | -    | -      | 0.2    |
| EDDS                           | -      | 0.25   | 0.4  | -    | 0.5    | 0.5    |
| PEG                            | 0.5    | 1.0    | 2.0  | 2.0  | 1.5    | -      |
| acetate salt                   | -      | -      | 0.3  | -    | 0.4    | 0.4    |
| soap                           | 1.5    | 2.0    | -    | -    | -      | 0.5    |
| Protease                       | 0.5    | 0.5    | 0.26 | 1.0  | 0.1    | 0.2    |
| Cellulase                      | 0.1    | -      | -    | 0.1  | 0.3    | 0.3    |
| Amylase                        | 0.4    | 0.5    | 0.2  | 0.4  | 1.0    | 0.1    |
| Lipase                         | 0.3    | -      | 0.5  | -    | -      | 0.5    |
| Photoactivated bleach<br>(ppm) | 15 ppm | 45 ppm | -    | -    | 20 ppm | 20 ppm |

|                 |      |      |     |     |      |     |
|-----------------|------|------|-----|-----|------|-----|
| PVNO/PVPVI      | -    | -    | 0.3 | 0.1 | -    | -   |
| Brightener      | 0.09 | 0.02 | 0.1 | -   | 0.06 | 0.1 |
| Perfume         | 0.3  | 0.3  | 0.3 | 0.4 | 0.4  | 0.4 |
| Suds suppresser | 0.1  | 0.1  | 0.5 | -   | 0.3  | 0.3 |

Claims

1. An effervescence particle comprising an anhydrous perborate salt intimately mixed with a diluent, with the proviso that the diluent is not sodium perborate monohydrate.
2. An effervescence particle according to claim 1 obtainable by a process comprising the steps of
  - a) obtaining an anhydrous perborate salt;
  - b) in a mixing step, intimately mixing the anhydrous perborate salt with a diluent.
3. An effervescence particle according to claim 2 wherein in the mixing step the diluent or part thereof is in liquefied form, preferably a melt.
4. An effervescence particle according to claim 2 or 3 wherein the diluent comprises a compound which has a melting point between 40°C and 200°C, preferably comprising a polymeric organic compound or a anionic or nonionic surface active compound, preferably selected from the group comprising anionic alkyl sulphates or sulphonates, nonionic alkoxyated amides, alkyl esters of fatty acids, or alkoxyated alcohols.
5. An effervescence particle according to claim 1 or claim 2 obtainable by a process comprising the steps of
  - a) in a mixing step, intimately mixing a hydrated perborate salt, preferably a perborate monohydrate salt, with a diluent to obtain an intimate mixture ;
  - b) dehydration of the intimate mixture of step a).

6. An effervescence particle according to claim 5, wherein the diluent comprises an organic or inorganic salt or acid, preferably a sulphate, borate, carbonate, phosphate or halide salt, or a polymeric, oligomeric or monomeric carboxylic or polycarboxylic acid or salt thereof.
7. An effervescence particle according to any of claims 1 to 6, wherein the anhydrous perborate salt is a sodium or potassium salt.
8. An effervescence particle according to any of claims 1 to 7 in the form of an agglomerate, compacted granule or extrudate.
9. An effervescence particle according to any of claims 1 to 8 wherein the total amount of the perborate salt, comprising the anhydrous perborate salt and optionally a hydrated perborate salt, is from 3% to 80%, preferably from 5% to 60% by weight of the component, whereby the amount of sodium perborate monohydrate is preferably less than 15%, preferably less than 10% by weight of the total amount of perborate salt.
10. A laundry or dish washing detergent composition comprising an effervescence particle according to any of claims 1 to 9, preferably a solid composition in the form of a granule, tablet, extrudate or bar, whereby the component is present as part of a component of the composition or as separate component of the composition, preferably comprising one or more surfactants.
11. A process for making an effervescence component according to claims 1 to 4, comprising the steps of
  - a) obtaining an anhydrous perborate salt;

- b) in a mixing step, intimately mixing the anhydrous perborate salt of a) with a diluent.
12. A process for making an effervescence component according to claims 5 or 6, comprising the steps of
- a) in a mixing step, intimately mixing a hydrated perborate salt, preferably a perborate monohydrate salt, with a diluent to obtain an intimate mixture;
- b) dehydration of the intimate mixture of step a).
13. Use of an effervescence component in a detergent composition according to any of claims 1 to 9 for improvement of the dispensing or dissolution of the detergent composition into the washing water.



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

|  |           |   |
|--|-----------|---|
| <b>(51) International Patent Classification <sup>6</sup> :</b><br><b>C01B 15/00, C11D 3/00</b>   | <b>A3</b> | <b>(11) International Publication Number:</b> <b>WO 99/58444</b><br><b>(43) International Publication Date:</b> 18 November 1999 (18.11.99)   |
| <b>(21) International Application Number:</b> PCT/US99/10008<br><b>(22) International Filing Date:</b> 7 May 1999 (07.05.99)<br><br><b>(30) Priority Data:</b><br>9809733.0                      8 May 1998 (08.05.98)                      GB<br><br><b>(71) Applicant (for all designated States except US):</b> THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US).<br><br><b>(72) Inventors; and</b><br><b>(75) Inventors/Applicants (for US only):</b> BAILLELY, Gérard, Marcel [FR/GB]; Whitebridge Park, 16 Saxilby Drive, Gosforth, Newcastle upon Tyne NE3 5LS (GB). HARTSHORN, Richard, Timothy [GB/GB]; 139 Dene Road, Wylam, Northumberland NE41 8EY (GB). JANSEN, Judit, Ester [NL/GB]; 27 Shaftesbury Grove, Heaton, Newcastle upon Tyne NE6 5FA (GB).<br><br><b>(74) Agents:</b> REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217-1087 (US). |           | <b>(81) Designated States:</b> JP, US, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).<br><br><b>Published</b><br><i>With international search report.</i><br><i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i><br><br><b>(88) Date of publication of the international search report:</b><br>9 March 2000 (09.03.00) |
| <b>(54) Title:</b> EFFERVESCENCE COMPONENT<br><br><b>(57) Abstract</b><br><br>The invention provides an effervescence particle comprising an anhydrous perborate salt intimately mixed with a diluent, with the proviso that the diluent is not sodium perborate monohydrate. The invention also relates to processes for making the particle and to detergent compositions comprising such a particle. The invention also relates to the use of such an effervescence particle in a detergent composition for improvement of the dispensing or dissolution of the detergent composition into the washing water or for improvement of the delivery of detergent actives of the composition to the wash or for improved sudsing.  |           |   |

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# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 99/10008

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 C01B15/00 C11D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C01B C11D

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Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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☐ Further documents are listed in the continuation of box C.

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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